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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/GB96/01252 (22) International Filing Date: 24 May 1996 (24.05.96) (30) Priority Data: 9510620.9 25 May 1995 (25.05.95) GB (71) Applicant (for all designated States except US): BICC PUBLIC LIMITED COMPANY [GB/GB]; Devonshire House, Mayfair Place, London W1X 5FH (GB). (71) Applicant (for US only): KATZ, Maurice (legal representative of the deceased inventor) [GB/AU]; 2 Rosslyn Court, Seaford, VIC 3198 (AU). (72) Inventor: PANASIUK, Leon (deceased). (72) Inventors; and (75) Inventors/Applicants (for US only): VERNE, Stefan [GB/GB]; 3 Brunswick Gardens, London W5 1AP (GB). BIELSTEIN, George, Paul, Richard [GB/GB]; 14 Temple Sheen, London SW14 7RP (GB). (74) Agent: POOLE, Michael, John; BICC Patents & Licensing Dept., Quantum House, Maylands Avenue, Hemel Hempstead, Hertfordshire HP2 4SJ (GB).			(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  Published With international search report.
(54) Title: POLYMER COMPOSITIONS AND ELECTRIC CABLES			
(57) Abstract  An environmentally desirable composition with sufficient electrical stability to be suitable for making the dielectric (insulation) of a medium-voltage cable comprises, in parts by weight: polymer base, of which at least 50 parts are ethylene copolymer rubber and the balance, if any, polyethylene 100, mineral filler of which at least half is calcined clay 40-120, surface treatment agent for the filler 1-6, zinc oxide 10-30, lead compounds not to exceed 1 calculated as element and appropriate amounts of conventional ingredients comprising an antioxidant, a curing system and a processing aid.			

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### Polymer Compositions and Electric Cables

This invention relates to polymer compositions for use in electric cables, and more particularly as dielectric (insulation) in "medium voltage" power cables in which the dielectric is bounded by a conductor screen and a core screen both of conductive polymeric material; it also relates to the cables in which they are used.

More particularly, it relates to compositions based on ethylene copolymer rubbers, which may be defined for certainty in the context of this application as hydrocarbon copolymers of ethylene with at least one other alkene monomer having from 3 to 10 carbon atoms which copolymers have rubbery elastic characteristics.

Until recently, only two ethylene copolymer rubbers have been widely used in such compositions, namely EPR, in which the major repeating units are ethylene and propylene, and EPDM, in which a minor proportion of a diene monomer (such as norbornadiene) is present in addition to ethylene and propylene. More recently, the development of "single-site" catalysts has made available additional ethylene copolymer rubbers which appear to be based predominantly on ethene and octene or on ethylene and hexene.

The service life of medium-voltage cables is in many cases limited by the long term diminution of electric strength of the dielectric which is now attributed to the formation of water-trees (or electric trees, if water is absent), and current EPR formulations generally contain an oxide of lead in an amount corresponding to several phr (parts per hundred of polymer) of lead, calculated as element, as the means of securing a sufficient stability of electrical strength (a tree suppressant). Although the amount of lead used in electric cables is small compared with other usages, it is now considered desirable to eliminate any

avoidable source of lead in contact with the environment.

Zinc oxide is a conventional constituent of EPR- and EPDM-based compositions, used at a level around 5phr as a curing aid or pH stabiliser; but it has not been perceived as having a useful effect on electrical stability.

Partly as a result of a review of unpublished experiments carried out in our laboratories by the late Leon Panasiuk under the supervision of Dr George P R Bielstein and Mr Stefan Verne between about 1964 and 1967, we have now recognised that higher levels of zinc oxide lead to useful levels of electrical stability under wet conditions, even in compositions in which the content of lead compounds is below 1%.

The compositions in accordance with the present invention comprise, in parts by weight:

	Polymer base, of which at least	
	50 parts are ethylene copolymer	
	rubber and the balance, if any,	
	polyethylene	100
20	Mineral filler of which at least	
	half is calcined clay	40-120
	Surface treatment agent for	
	the filler	1-6
	Zinc oxide	10-30
25	Lead compounds	not to exceed 1 calculated as element

and appropriate amounts of conventional ingredients comprising an antioxidant, a curing system and a processing aid.

The ethylene copolymer rubber may be an EPR or an EPDM, each of which may be either semi-crystalline or

amorphous provided that it has rubbery elastic properties. Ethylene copolymer rubbers made with "single-site" catalyst systems can also be used and may have advantages in some cases.

5 Preferably all the filler is calcined clay.

The surface treatment agent for the filler is preferably selected from silanes, siloxanes, metal salts of organic acids, and organic acids that are substantially insoluble in water. More than one surface treatment agent  
10 can be used. Silane surface treatment agents are preferred. The filler is preferably pre-treated with the surface treatment agent (or with one of them) but preferably the composition includes free surface treatment agent as well. Appropriate amounts will vary somewhat from one surface  
15 treatment agent to another, but will typically be in the range from about 1-2 parts pre-coated on the filler and about 1-5 parts added separately.

Preferred content of zinc oxide is about 15-25 parts; conventional grades can be used, but grades of higher purity  
20 are preferred.

Compositions based on semicrystalline EPR's or EPDM's preferably contain polyethylene, preferably in an amount in the range from 3 to 10 parts, as a processing aid; larger amounts, up to the limit of 50 parts, can be used for cost  
25 reduction and/or in the case of some amorphous EPR's or EPDM's to enable granulation of the composition.

From an environmental viewpoint, the content of lead compounds should be as small as possible, and ideally zero; however, a small amount of certain lead compounds may achieve  
30 a major improvement in electrical stability with much less environmental hazard than conventional EPR or EPDM

compositions for medium-voltage cables. In particular, there may be merit in using up to about 2 parts of lead stearate (that is up to about 0.54 parts calculated as element).

Any of the conventional antioxidants can be used in their usual amounts; we particularly prefer antioxidants of the poly-dihydroxyquinoline class, in amounts in the range 0.5 to 3 parts.

Conventional curing systems can be used; in particular, all the compositions of the invention can be cured with peroxides, optionally with poly-unsaturated co-agents such as triallylisocyanurate, or with peroxides and sulfur; compositions based on EPDM's can also be cured with conventional sulfur-based systems. No change in curing practice is expected to be needed.

Suitable processing aids, apart from those already mentioned, include paraffin wax in conventional amounts up to around 6 parts and hydrocarbon oils in amounts up to around 30 parts (varying from oil to oil).

#### EXAMPLES

Compositions as detailed below were prepared on a 12-inch (300mm) laboratory 2-roll mill; the polymer-base was plasticised and the other ingredients, except curing agents, added at a temperature controlled to be in the range 120-130°; the curing agents were added in a separate step on the "cold" mill. It should be noted that a few ingredients used in experiments around 30 years ago are no longer available.

#### Example 1

To 95 parts of an EPDM (Royalene® 201) and 5 parts of a polyethylene (WJG11) were added: 67 parts calcined clay (Whitetex®) and 48 parts talc (Mistron Vapour®) as additional mineral filler; 1 part vinyl tris(2-methoxyethoxy) silane

(Silane Al72®) as surface treatment agent; 14 parts zinc oxide; ; 1 part of polymerised trimethyldihydroquinoline (Flectol H®) and 1.3 parts of *N*-nitroso-*N*-methyl-*p*-nitrosoaniline as antioxidants; a curing system comprising 5 1 part dicumyl peroxide and 1 part *N,N'*(1,3-phenylene) dimaleimide; and 9.5 parts of Esso® Oil D and 5 parts of paraffin wax as processing aids. After press cure for 45 minutes at 160°, the composition had a tensile strength of 8.48MPa (1230 psi), elongation (meaning elongation at break) 10 of 250% and volume resistivity of  $4.9 \times 10^{15}$  ohm.cm.

In preliminary ageing tests in air at 150°C, the composition showed first signs of brittleness after about 35 days.

Electrical aging tests were also carried out using the 15 material extruded to a minimum radial thickness of 0.76mm (0.030 inch) onto a strand comprising three tinned copper wires each 0.74mm (0.029inch) in diameter. The insulated strand was immersed in water at 75°C and exposed continuously (except during measurements) to a voltage of 600V at 50Hz; at 20 intervals, insulation resistance was measured at 500V dc one minute after application of this voltage and specific inductive capacitance and power factor at 1.2kV 50Hz. Within one day's exposure, the volume resistivity had fallen to  $1.5 \times 10^{14}$  ohm.cm, but this then remained substantially 25 unchanged (readings ranged from  $1.0$  to  $2.6 \times 10^{14}$ ) up to and including the 59th week. Deterioration was detectable in about weeks 60 to 70, when readings ranged from 0.49 to  $1.0 \times 10^{14}$  ohm.cm, and gradually increased with the readings falling below  $0.1 \times 10^{14}$  ohm.cm for the first time at week 74 30 and reaching  $0.014 \times 10^{14}$  when the tests were discontinued at



120 weeks. Specific inductive capacitance varied by no more than 5% from its value after one day's exposure until week 78, and had changed by only about 8% at the end of 120 weeks. Power factor changed little during the tests; after 5 one week, it was in the range 6-7.2%, where it remained for 19 weeks; it then slightly reduced, falling below 3% at week 32 and remaining between 2.0 and 3.0 until week 57, when a slow rise was observed, reaching 4% at week 94, 4.8 at week 112 and then rising more rapidly to 5.3 at week 114, 6.8 at 10 week 118 and 7.7 at the end of the test at 120 weeks.

#### Example 2

This formulation was similar to Example 1 but 1 part of oleic acid was added as an additional surface treatment agent for the filler. After press cure for 45 minutes at 160°, the 15 composition had a tensile strength of 7.07MPa (1025psi), elongation of 390% and volume resistivity of  $13 \times 10^{15}$  ohm.cm. This composition showed first signs of brittleness in the preliminary air ageing test after 48 days at 150°C. The volume resistivity in the wet electrical 20 ageing test remained in the range  $0.9$  to  $2.3 \times 10^{14}$  ohm.cm from about 3 to 43 weeks and then slowly declined, reaching  $0.01 \times 10^{14}$  ohm.cm at about 52 weeks and  $0.007 \times 10^{14}$  ohm.cm when the test was discontinued at 71 weeks. Specific inductive capacitance changed by 5.2% within 1 week and then 25 increased slowly and fairly steadily to have changed about 16% after 59 weeks; lower change values (in the range 5-10%) were obtained in weeks 61-71, but may be suspect.

#### Example 3

To 100 parts of an experimental grade of EPDM (DuPont® ECD- 30 618-60) were added 120 parts of a proprietary silane-

pretreated calcined clay (Translink® 37), 1 part of stearic acid as an additional surface treatment agent for the clay, 20 parts of zinc oxide, 1 part of a primary aromatic amine antioxidant (LD-382-30), a curing system comprising 1.25 parts sulfur, 3 parts of a proprietary mixture (EPTAC.2) comprising equal weights of zinc dimethyl dithiocarbamate and tetra-methyl thiuram disulfide, 2 parts of 2-mercapto-benzothiazole and 1.3 parts of 75%-active tellurium diethyl dithiocarbamate (PTD-75) and as processing aids 30 parts of Sunpar® oil 2280 and 3 parts paraffin wax. After press cure for 45 minutes at 160°, the composition had a tensile strength of 4.4MPa (645psi) and an elongation of 430%. This composition did not show signs of brittleness in the air ageing test until the 60th day.

The composition was made into an insulated wire sample and tested as before, and the insulation resistance observed remained in the range  $2.6$  to  $4.9 \times 10^{14}$  ohm.cm throughout 39 weeks of observation; the specific inductive capacity changed gradually over about 21 weeks and then remained fairly stable with the change remaining in the range 7.6 to 10.2% for the remainder of the test; power factor was essentially constant in the range 0.9 to 1.2% throughout the test.

#### Example 4

This was identical with Example 1, except that 1 part of lead stearate was added. After press cure for 45 minutes at 160°, the composition had a tensile strength of 6.4MPa (925psi), elongation of 515% and volume resistivity of  $9.2 \times 10^{15}$  ohm.cm.

In this case, the volume resistivity remained in the range 1.1 to  $3.6 \times 10^{14}$  ohm.cm throughout the 120-week period

of the test in water, with values in the upper part of the range (over 2.5) observed mostly in the period from 34 to 68 weeks. Measurements of increase in specific inductive capacitance showed no very clear pattern, but were mostly below 5% for the first 64 weeks and mostly in the range 5-10% in weeks 65-120. Power factors observed were all in the range from 1.7 to 2.8, with only about five readings over 2.0% after week 30.

#### Example 5

10 The composition of this example comprises 95 parts of a semicrystalline EPR (Nordel® 2722), 5 parts of a low-density polyethylene of Melt Index 2.0, 60 parts of surface-treated calcined clay (Translink® 37), 1 part of vinyl tris(2-methoxy ethoxy) silane, 20 parts zinc oxide, 5 parts paraffin wax, 15 1.5 parts antioxidant (Flectol® H) and 2.5 parts of dicumyl peroxide (Dicup®).

#### Example 6

The composition of this example comprises 100 parts of an amorphous EPR (Nordel® 2522), 60 parts of surface-treated 20 calcined clay (Translink® 37), 1 part of vinyl tris(2-methoxy ethoxy) silane, 20 parts zinc oxide, 5 parts paraffin wax, 1.5 parts antioxidant (Flectol® H) and 2.5 parts of dicumyl peroxide (Dicup®).

#### Example 7

25 The composition of this example comprises 100 parts of an semicrystalline EPR (Nordel® 2722), 60 parts of surface-treated calcined clay (Translink® 37), 1 part of vinyl tris(2-methoxy ethoxy) silane, 20 parts zinc oxide, 1.5 parts paraffin wax, 1.5 parts antioxidant (Agerite® MA) and 2.5 30 parts of dicumyl peroxide (Dicup®).

Example 8

This was identical with Example 7 except that the clay was a non-commercial untreated one, identical with Translink 37 except for the omission of the surface treatment normally applied to it.

Example 9

This was similar to Example 7 except that 40 parts of a polyethylene with Melt Index 2.0 and density about 0.92 was substituted for an equal number of parts of the EPR and that the content of zinc oxide was only 10 parts.

Comparison Examples A and B

These formulations were similar to Example 1 except that the content of zinc oxide was only 5 parts and that there were minor differences in the antioxidant and curing systems: in both cases, half of the Flectol® H was replaced with an equal weight of a third antioxidant, zinc-2-mercapto-benzimidazolate; the curing systems comprised:

Comparison Example A	sulfur (1 part);
	tellurium diethyl dithiocarbamate
20	(Tellurac®) (1.5 parts, 80% active);
	dipentamethylene thiuram
	tetrasulfide (Tetrone® A)
	(1.5 parts); and
	zinc dimethyl dithiocarbamate
25	(0.5 part) -
Comparison Example B	dicumyl peroxide (Dicup®) (3 parts)
	and sulfur (0.3 part).

Both these comparison examples were rejected on the basis of preliminary screening tests in air at 150°C, and were not subjected to electrical stability tests; comparison Example A exhibited brittleness in this test at 11 days and Comparison

Example B surface degradation at 6 days.

Comparison Example C

This was identical with Example 1 except that a conventional 3 parts of litharge was added. The composition showed  
5 embrittlement in the air ageing test at 48 days (rather better than Example 1, but no better than wholly lead-free Example 2). In the electrical stability test, the volume resistivity slowly rose from an initial value of about  $1.2 \times 10^{14}$  ohm.cm to reach  $3 \times 10^{14}$  ohm.cm about week 36,  
10 remaining fairly stable to week 60 and then declining steadily to about  $0.03 \times 10^{14}$  ohm.cm at the end of 120 weeks.

Specific inductive capacitance changes for this composition were nearly all negative, ranging from +0.4 (at one week) to -7.5 (at 66 weeks) without any clear pattern. Power factor  
15 was similar almost constant, ranging from 1.1 to 4.7.

Comparison Example D

This is similar to Example 3 except for the addition of 4 parts of lead dioxide and the use of a different curing system consisting of 1 part dicumyl peroxide and 1 part  
20 N,N'-(1,3 phenylene) dimaleimide. This composition showed signs of surface degradation in the air aging test after 34 days. In the electrical stability test, the volume resistivity was higher than for Example 3, ranging from 4.4 to  $6.7 \times 10^{14}$  ohm.cm over weeks 1-39, but there was no  
25 noticeable difference in stability; changes in specific inductive capacitance were also smaller (not over 4.3%) with the highest values (over 2.0%) observed in weeks 6 to 33. Power factor was substantially constant and a little better than for Example 3, with values from 0.4 to 0.6% recorded.

## CLAIMS

- 1 A composition suitable for making the dielectric of a medium-voltage electric cable comprising, in parts by weight:
- |   |  |
|---|--|
| Polymer base, of which at least                                   |  |
| 5 50 parts are ethylene copolymer rubber and the balance, if any, |  |
| polyethylene  | 100                                      |
| Mineral filler of which at least                                  |  |
| half is calcined clay   | 40-120                                   |
| 10 Surface treatment agent for the filler                         | 1-6                                      |
| Zinc oxide  | 10-30                                    |
| Lead compounds  | not to exceed 1<br>calculated as element |
- 15 and appropriate amounts of conventional ingredients comprising an antioxidant, a curing system and a processing aid.
- 2 A composition as claimed in claim 1 in which the ethylene copolymer rubber is an EPR or an EPDM.
- 20 3 A composition as claimed in claim 1 in which the ethylene copolymer rubber is one made with "single-site" catalyst system.
- 4 A composition as claimed in any one of claims 1-3 in which all of the filler is calcined clay.
- 25 5 A composition as claimed in any one of claims 1-4 in which the surface treatment agent for the filler is selected from silanes, siloxanes, metal salts of organic acids, and organic acids that are substantially insoluble in water.
- 6 A composition as claimed in any one of claims 1-5 in
- 30 which the filler is pre-treated with the surface treatment agent.

- 7 A composition as claimed in claim 6 which also includes free surface treatment agent.
- 8 A composition as claimed in any one of claims 1-7 in which the content of zinc oxide is about 15-25 parts.
- 5 9 A composition in accordance with any one of claims 1-8 based on a semicrystalline EPR or EPDM and containing 3 to 10 parts polyethylene.
- 10 A lead-free composition in accordance with any one of claims 1-9.
- 10 11 A composition in accordance with any one of claims 1-9 comprising up to about 2 parts of lead stearate.
- 12 A medium-voltage electric cable having dielectric made from the composition claimed in any one of claims 1-11.
- 13 A lead-free composition suitable for making the
- 15 dielectric of medium-voltage cable substantially as described with reference to either Example 5 or Example 6.
- 14 A lead-free composition suitable for making the dielectric of medium-voltage cable substantially as described with reference to any one of Examples 7-9.

## INTERNATIONAL SEARCH REPORT

International Application No  
PC/GB 96/01252

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 H01B3/44

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 H01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 042 440 A (DAINICHI NIPPON CABLES LTD) 30 December 1981 see page 20, line 11 - line 17 additive 29 see page 21 see page 23 - page 24; examples 1-19 see page 37; example 38 see claims 1-19	1-14
X	FR 2 344 613 A (ALCAN RES & DEV) 14 October 1977 see claims 1-18; examples 15-19	1-14
A	US 4 204 024 A (PYE RICHARD T) 20 May 1980 see claims 1-12	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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